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TECHNICAL NOTE

No. 1102

THE CRYSTAL STRUCTURE AT ROOM TEMPERATURE OF EIGHT

FORGED HEAT-RESISTING ALLOYS

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SUMMARY

In order to determine the crystal structure of some currently used heat-resisting alloys, a preliminary investigation of eight leading forged alloys - S816, S590, Gamma Columbium, Hastelloy B, 16-25-6, 19-9 DL, Nimonic 80, and N155 (low carbon) - was conducted by X-ray diffraction methods. The predominant phase in each alloy was found to be a solid solution of the chief alloying elements. The crystal structure of the solid solution was the face-centered cubic type. Alloys S816, S590, and Gamma Columbium, which contained the largest percentages of columbium, were found to show diffraction lines from a second phase that is believed to be columbium carbide, CbC.

INTRODUCTION

Current gas turbines and jet-propulsion engines are greatly limited in performance by the maximum temperatures at which the component metal parts may operate without failure. Such failures may occur in the form of actual fractures, intercrystalline corrosion, distortion due to temperature gradients and centrifugal stresses, and excessive elongation due to creep. The suitability of a metal part for withstanding highly adverse operating conditions, such as those found in gas turbines and jet-propulsion engines, is often largely determined by its crystal structure.

X-ray diffraction is one of the most satisfactory methods for the study of crystal structures. Data obtained at the NACA Cleveland laboratory by X-ray diffraction methods in a preliminary

investigation of the crystal structure of eight forged heat-resisting alloys are presented. Interplanar spacings and intensities of diffracted lines are given as well as lattice constants, which were calculated from interplanar-spacing data.

APPARATUS AND TEST PROCEDURE

Chemical compositions of the eight forged alloys investigated (S816, S590, Gamma Columbium, Hastelloy B, 16-25-6, 19-9 DL, Nimonic 80, and N155 (low carbon)) are listed in table 1. The compositions, with the exception of those for Hastelloy B and 16-25-6, are mill analyses supplied by the manufacturers of the alloys. The actual analyses for Hastelloy B and 16-25-6 were not available but a typical analysis from reference 1 for these two alloys is presented. The alloys studied include some of the leading compositions currently used at temperatures above 1200° F (references 1 and 2). Specimens of all of the alloys with the exception of Hastelloy B were taken from either hot-rolled bars or rounds. Hastelloy B specimens were taken from filings removed from a finished forging of the alloy.

X-ray diffraction patterns of the eight alloys were made with filtered Co K α radiation in a Debye-Scherrer powder camera, which was 143.2 millimeters in diameter. The X-ray beam was collimated by slits measuring 0.020 by 0.300 inch. A specimen from each alloy was mounted in the camera and oscillated through 20° in the X-ray beam. The solid metal specimens were etched to a depth of about 0.010 inch to remove cold-worked surface material produced by the sectioning process. The filings of Hastelloy B were annealed at 1000° F for 30 minutes to remove cold work.

Diffraction patterns of the same specimens of the alloys were also made with unfiltered Fe K α and K β radiation in a Sachs type back-reflection camera at a film-specimen distance of 5 centimeters. The exact distance was determined for each pattern by calibration with a standard specimen of pure gold.

Intensities of the lines of each diffraction pattern were measured with a Knorr-Albers microphotometer. Line spacings were measured on the film to the nearest 0.05 millimeter. All measurements of line spacings were corrected for film shrinkage.

RESULTS AND DISCUSSION

Interplanar-spacing values and intensities of the lines appearing in the Debye-Scherrer diffraction pattern of each alloy are tabulated in table 2. At room temperature each of the eight alloys gave a diffraction pattern of five strong lines with the sequence characteristic of the face-centered cubic crystal structure. Experimental data (reference 3) indicate that this type of crystal structure shows lower values of creep rate than the body-centered cubic type, which occurs in some chromium-iron alloys. The positions of the lines in diffraction patterns of the alloys were found to shift proportionately with differences in composition of the alloys; this shift indicates that a solid solution rather than a mixture exists between the main alloying elements. The phases found at room temperature may quite possibly represent only a metastable condition because of the relative immobility of the metal atoms at room temperature.

The lattice constant a for each alloy was calculated from its back-reflection pattern using the relation

$$a = \lambda \frac{\sqrt{h^2 + k^2 + l^2}}{2 \sin \theta}$$

where

λ wave length of X-rays being scattered, A

θ angle formed by incidence of primary X-ray beam on reflecting atomic plane

h, k, l Miller indices of reflecting plane

In each case the $K\alpha_1$ reflection from the (222) planes was used for the calculation of the lattice constant. The lattice constants determined by the Sachs type back-reflection camera for the alloys are presented in table 3 and are believed to be accurate to ± 0.0005 A. Lattice constants a and c of the metals present in relatively large amounts in the alloys and their crystal structures are given in table 4. Data for this table were taken from reference 4 (pp. 552-554).

In diffraction patterns of alloys S816, S590, and Gamma Columbium, lines of relatively low intensities were noted in addition to all possible lines from the face-centered cubic phase 1 (table 2).

A second phase, or compound, appears therefore to occur in these three alloys. Of the eight alloys investigated, alloys S816, S590, and Gamma Columbium are distinguished from the other five by the relatively large percentages of columbium they contain. Columbium as an alloying element is noted for its carbide-forming tendency and for that purpose is often added to alloys containing chromium (reference 5). Calculated interplanar spacings d and relative intensities I/I_1 for columbium carbide, CbC, are listed in table 5 together with observed interplanar spacings and intensities of the second phase, or compound. According to reference 6, the lattice constant of CbC is 4.40; this value was used in calculating the interplanar spacings. Calculations of intensities made use of the structure factor, the multiplicity factor, and the Lorentz and polarization factors, as described in reference 4 (pp. 524-541).

An excellent agreement between the d values of the six lines of the second phase and the six strongest lines of CbC is shown in table 5. It is not known why certain lines from the second phase were missing in patterns of one or two of the three alloys showing the second phase. These lines may not have been discernible because of the general fogged background of the patterns, which was found to vary for different alloys. Only fair agreement exists between average intensities of the unknown lines and calculated intensities of CbC. In most cases the observed diffraction lines of the second phase were so weak that quantitative evaluations of the intensities of the lines were subject to relatively large errors. Columbium carbide, CbC, appears to exist, however, in alloys S816, S590, and Gamma Columbium in amounts great enough to be detected by X-ray diffraction methods.

SUMMARY OF RESULTS

The following results have been obtained by applying X-ray diffraction methods to eight forged heat-resisting alloys, S816, S590, Gamma Columbium, Hastelloy B, 16-25-6, 19-9 DL, Nimonic 80, and NL55 (low carbon):

1. The predominant phase occurring in each alloy was a solid solution of the alloying elements present in relatively large amounts. The crystal structure of the solid solution was the face-centered cubic type.

2. Alloys S816, S590, and Gamma Columbium, which contain the largest percentages of columbium, showed diffraction lines from a second phase that is believed to be columbium carbide, CbC.

Aircraft Engine Research Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, April 10, 1946.

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2. Freeman, J. W., Rote, F. B., and White, A. E.: High Temperature Characteristics of 17 Alloys at 1200° and 1350° F. NACA ACR No. 4C22, 1944.
3. Anon.: Wrought Heat Resisting Steels. Metals Handbook, Am. Soc. Metals, 1939 ed., pp. 544-549. (Prepared for the Subcommittee on Wrought Heat Resisting Steels, Am. Soc. Metals, by H. D. Newell and John J. B. Rutherford.)
4. Barrett, Charles S.: Structure of Metals. McGraw-Hill Book Co., Inc., 1943.
5. Thum, Ernest E.: The Book of Stainless Steels. Am. Soc. Metals, 2d ed., 1935, pp. 307-308, 389.
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TABLE 1. - COMPOSITIONS OF EIGHT FORGED HEAT-RESISTING ALLOYS STUDIED BY X-RAY DIFFRACTION METHODS

[Typical compositions of Hastelloy B and 16-25-6 from reference 1; all other values supplied by the manufacturers]

Alloy	Chemical composition, percent												
	C	Si	Mn	Cr	Ni	Co	Mo	W	Cb	Ti	Al	Fe	Other
S816	0.36	0.19	0.72	18.40	20.23	45.63	3.72	4.23	3.72	----	----	3.48	-----
S590	.47	.82	1.35	19.40	19.07	19.26	4.03	4.00	3.87	----	----	Bal.	-----
Gamma Columbium	.40	.58	.92	15.18	24.97	-----	4.17	----	2.16	----	----	Bal.	Cu 0.06
Hastelloy B	.12	.52	1.20	.27	60.83	-----	27.10	----	----	----	----	----	-----
16-25-6	.06	.85	1.54	16.55	25.2	-----	6.46	----	----	----	----	----	N ₂ .07
19-9 DL	.29	.50	1.41	18.59	9.04	-----	1.19	1.40	.45	0.29	----	Bal.	Cu .12
Nimonic 80	.02	.62	.77	20.77	74.00	-----	-----	----	----	2.50	0.57	.66	Cu .06
N155 (low carbon)	.16	.50	1.65	21.06	19.79	20.25	3.14	1.68	1.13	----	----	Bal.	N ₂ .08

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TABLE 2. - X-RAY DIFFRACTION DATA ON EIGHT FORGED HEAT-RESISTING ALLOYS

[Filtered Co K α radiation was used to obtain diffraction patterns;
 d , interplanar spacing Å; I , peak intensity of a diffraction line;
 I/I_1 , relative intensity of a diffraction line where I_1 is the
intensity of the strongest line]

Two-phase alloys											
S816				S590				Gamma Cromium			
Phase 1		Phase 2		Phase 1		Phase 2		Phase 1		Phase 2	
d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
----	----	2.55	0.11	----	----	----	----	----	----	----	----
----	----	2.20	.08	----	----	----	----	----	----	----	----
2.05	1.00	----	----	2.07	0.50	----	----	2.07	1.00	----	----
1.78	.39	----	----	1.79	.44	----	----	1.79	.46	----	----
----	----	1.56	.10	----	----	1.57	0.17	----	----	1.56	0.05
----	----	1.33	.07	----	----	1.34	.22	----	----	1.34	.04
1.26	.44	----	----	1.27	.72	----	----	1.27	.37	----	----
1.07	.53	----	----	1.08	1.00	----	----	1.08	.54	----	----
1.03	.19	----	----	1.03	.56	----	----	1.03	.29	----	----
----	----	----	----	----	----	1.01	.04	----	----	----	----
----	----	.989	.04	----	----	.989	.10	----	----	.989	.02
One-phase alloys											
Hastelloy B		16-25-6		19-9 DL		Nimonic 80		N155 (low carbon)			
d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
2.08	1.00	2.08	0.73	2.07	1.00	2.05	0.72	2.07	1.00		
1.79	.42	1.80	.70	1.79	.78	1.77	.61	1.79	.61		
1.27	.31	1.27	.86	1.26	.57	1.25	.50	1.26	.33		
1.08	.89	1.08	1.00	1.08	.70	1.07	1.00	1.08	.69		
1.04	.10	1.04	.50	1.03	.44	1.02	.39	1.03	.33		

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TABLE 3. - LATTICE CONSTANT a OF EIGHT FORGED HEAT-RESISTING
ALLOYS CALCULATED FROM INTERPLANAR-SPACING DATA

Alloy	Lattice constant a (A) (a)
S816	3.5733
S590	3.5928
Gamma Columbium	3.5932
Hastelloy B	3.5915
16-25-6	3.5975
19-9 DL	3.5918
Nimonic 80	3.5583
N155 (low carbon)	3.5867

^aAccurate to ± 0.0005 A.

TABLE 4. - CRYSTAL STRUCTURE AND LATTICE CONSTANTS OF METALS PRESENT
IN RELATIVELY LARGE AMOUNTS IN EIGHT FORGED HEAT-RESISTING ALLOYS
[Data from reference 4, pp. 552-554]

Metal	Crystal structure	Lattice constants (A)	
		a	c
α -Chromium	Body-centered cubic	2.8786	4.072
α -Cobalt	Hexagonal close-packed	2.507	
β -Cobalt	Face-centered cubic	3.545	
α -Iron	Body-centered cubic	2.8610	
γ -Iron	Face-centered cubic	3.564	
β -Nickel	Face-centered cubic	3.5169	

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TABLE 5. - COMPARISON OF AVERAGE INTERPLANAR SPACINGS AND
INTENSITIES OF THE SECOND PHASE FOUND IN ALLOYS S816, S590
AND GAMMA COLUMBIUM WITH CALCULATED INTERPLANAR SPACINGS
AND INTENSITIES OF COLUMBIUM CARBIDE, CbC

[d, interplanar spacing, A; I, peak intensity of
diffraction line; I/I_1 , relative intensity of
diffraction line where I_1 is intensity of
strongest line]

Phase 2		Columbium Carbide, CbC	
d	I/I_1	d	I/I_1
2.55	1.00	2.54	1.00
2.20	.73	2.20	.70
1.56	1.00	1.56	.43
1.34	1.00	1.33	.38
-----	-----	1.27	.16
-----	-----	1.10	.10
1.01	.36	1.01	.34
.989	.46	.984	.52

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